# A STUDY OF THE CURVATURE OF A THICK ALN FILM GROWN ON A TRENCH-PATTERNED α-$Al_2O_3$ TEMPLATE USING X-RAY DIFFRACTION

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**Abstract -** In this article a method using X-ray diffraction for determining the crystallographic curvature of a thick AlN crystalline film epitaxially grown on a periodically trench-patterned α-$Al_2O_3$ template by the hydride vapor phase epitaxy method was studied. A series of X-ray rocking curve measurements for AlN 0002 reflection was taken at different positions across the surface of the thick AlN epitaxial film along the $[1\bar{1}00]$ direction. We introduced a model for determining the crystallographic curvature and the curvature radius from X-ray diffraction results. The results clearly demonstrate that the crystallographic curvature of the film is convex along the $[1\bar{1}00]$ direction and the radius of crystallographic curvature of the thick AlN film is estimated to be 3.1 m.

**Key words -** Curvature; X-ray diffraction; AlN film; trench-patterned template; strain.

## 1. Introduction

Aluminum nitride (AlN) has attracted a significant amount of research interest in undeveloped fields such as deep ultraviolet (DUV) light emitting diodes, lasers, high frequency electronic devices… because of its wide bandgap energy of 6.2 eV [1-3]. AlN can alloy with gallium nitride (GaN) to form compounds such as $Al_xGa_{1-x}N$ (x = 0 ~ 1), which have potential applications in short wavelength optoelectronic devices. In addition, its properties such as high hardness, high thermal conductivity [4] and resistance to high temperatures and caustic chemicals [5] combined with a reasonable thermal match with Si and GaAs make AlN an attractive material for electronic packaging applications. However, due to difficulties of growing large-area bulk A1N crystals, the heteroepitaxial growth of thick AlN films on substrates such as α-$Al_2O_3$ and 6H-SiC via hydride vapor phase epitaxy (HVPE) in combination with metalorganic vapor phase epitaxy (MOVPE) is one of the more promising techniques being evaluated [6-8]. Unfortunately, lattice and thermal mismatches between AlN and its substrates are usually a major impediment to growing high quality crystalline AlN films because they induce the generation of crystallographic defects, residual strain and crystallographic curvature in such films during growth and cooling processes [9,10].

Several methods such as double crystal diffraction topography and two beam laser reflection techniques have been utilized in order to determine the crystallographic curvature of films epitaxially grown on substrates [11-13]. However, the experimental setup of these methods are complex because they require specific devices and configurations. In this study, we introduce a new method for determining the crystallographic curvature of the epitaxial films using rocking curve (RC) measurements of X-ray diffraction (XRD). The experimental setup of this method is available in any X-ray diffractometer.

## 2. Experiment

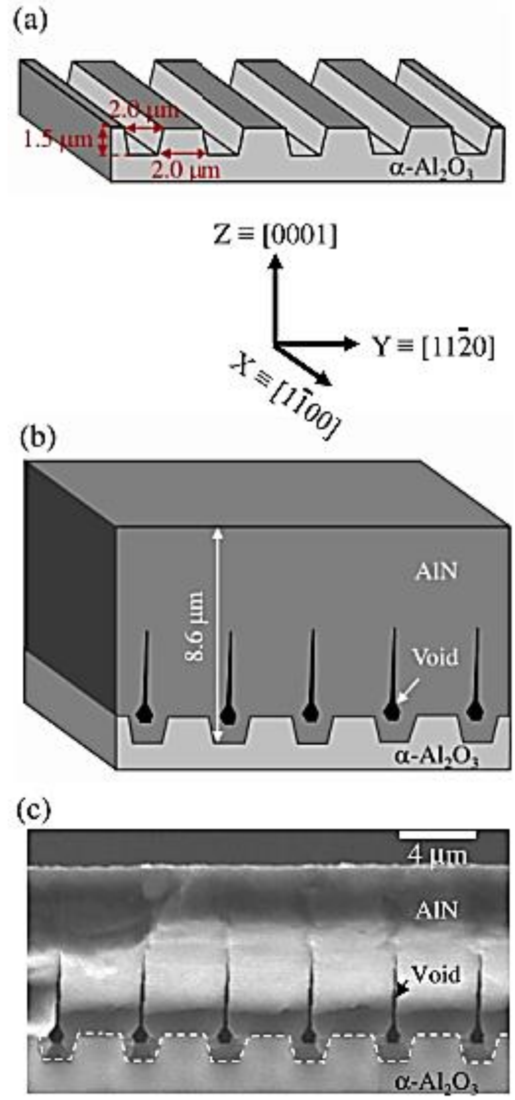


***Figure 1.*** *Schematic diagram of the sample fabrication process: First, (a) A trench-patterned α-$Al_2O_3$ template was fabricated from an α-$Al_2O_3$ substrate using the reactive ion etching technique; Then, (b) a thick AlN film was grown on the trench-patterned α-$Al_2O_3$ template using the HVPE method. (c) Cross-sectional SEM image of the thick AlN film grown on the trench-patterned α-$Al_2O_3$ template. The white dash line indicates the interface between the HVPE-grown AlN film and trench-patterned α-$Al_2O_3$ template*

The sample fabrication process is shown in Figure 1. The axes of X, Y and Z represent the directions of $[1\bar{1}00]$, $[11\bar{2}0]$ and [0001], respectively. First, as shown in Figure 1(a), a trench-patterned template was created on an α-$Al_2O_3$ substrate using the reactive ion etching technique. The trench direction was $[1\bar{1}00]$ and the pattern was periodic in the $[11\bar{2}0]$ direction. Trench depth was set at 1.5 µm while terrace and trench widths were both set at 2.0 µm. Then, as shown in Figure 1(b), an 8.6-µm-thick AlN film was grown on this template using a low-pressure HVPE system with infrared lamps as heaters. The growth pressure was 30 Torr and the growth temperature range was about 1400 – 1500°C. $NH_3$, Al, and HCl were used as source materials. $N_2$ and $H_2$ were used as carrier gases. A source of $AlCl_3$ was formed by the reaction of Al and HCl at 550°C in the source zone of the reactor. $AlCl_3$ was then reacted with $NH_3$ in the growth zone producing AlN layers on the trench-patterned $\alpha$-$Al_2O_3$ template. Figure 1(c) shows a cross-sectional scanning electron microscopy (SEM) image of the thick AlN film grown on the trench-patterned $\alpha$-$Al_2O_3$ template. Here, it can be observed that voids form tunnels running along the X direction over the trenches that were periodically arranged in the Y direction at 4-µm intervals.

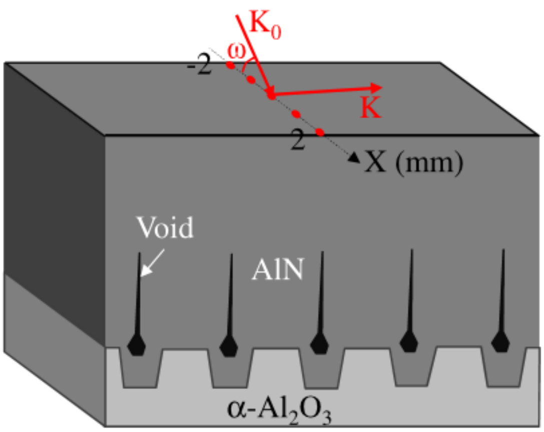


***Figure 2.*** *Schematic diagram of XRD from AlN (0002) planes. $K_0$ and K are the incident and diffracted X-ray beams, respectively. Red circles indicate sampling positions for RC measurements. ω is incident angle of X-ray beam to the film surface*

Figure 2 shows schematic diagram of XRD from AlN (0002) planes. In order to clarify the film curvature in the X direction, the X-ray incidence was selected so that the diffraction plane can be determined by the incident and diffracted vectors can be parallel to this direction. The film curvature in the X direction was clarified by taking a series of AlN 0002 RC measurements at different positions across the film surface along this direction with regular steps of 1 mm. The X-ray beam size was 0.1 mm × 0.1 mm. The X-ray wavelength and penetration depth were 0.15418 nm and 12.6 µm, respectively.

## 3. Results and discussion

Figure 3(a) shows the result of a series of 0002 RC measurements taken at different positions with 1-mm steps in the range of 4 mm along the X direction. It should be noted that each RC profile consists of a single peak forming a fairly uniform distribution along the X axis. This indicates that the crystalline morphology is fairly homogeneous in the $[1\bar{1}00]$ direction. This homogeneity leads to the remarkable curvature along the $[1\bar{1}00]$ direction as a form of macroscopic strain relaxation in this direction.

From the result in Figure 3(a), the incident angle $\omega$ at the maximum intensity in each RC profile was plotted as a function of the measured position. The result was shown in Figure 3(b). It is clearly observed that the incident angle $\omega$ linearly changes with the position along the X direction. As schematically shown in Figure 4(a), it can be determined that the curvature of the lattice planes in the AlN film is convex when an $\omega$-incident angle increase is observed by shifting the X-ray beam in the direction of X. In contrast, as shown in Figure 4(b), a concave curvature exists when an $\omega$-incident angle decrease is observed by shifting the X-ray beam in the direction of X. An inspection of the result shown in Figure 3(b) clarifies that the former is the case for the present AlN film. The convex film curvature in the $[1\bar{1}00]$ direction is due to the presence of the compressive strain in this direction [10, 14]. According to the model shown in Figure 5, the radius of curvature $R$ can be expressed in the form:

$$R = \frac{L}{\Delta\omega}.$$

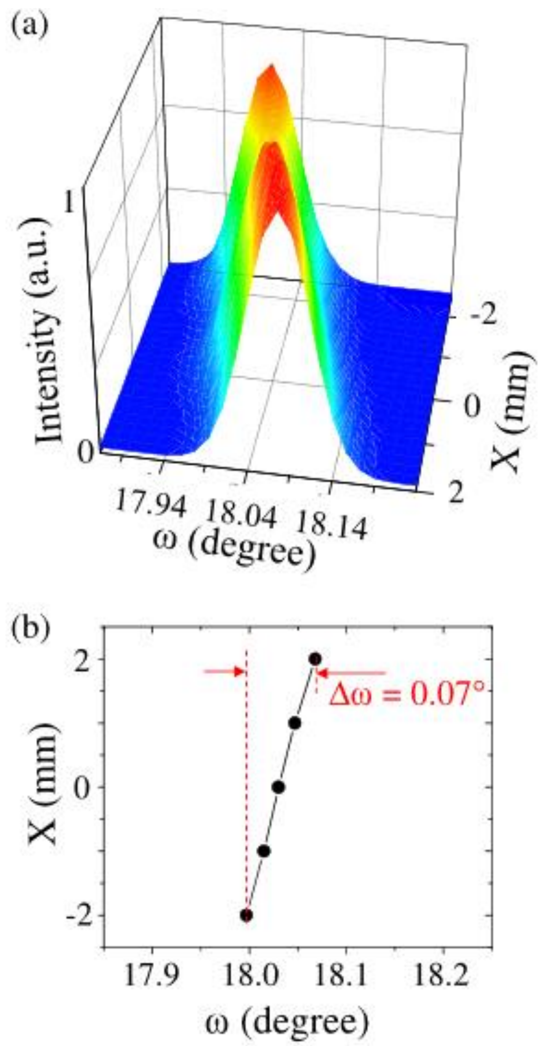


***Figure 3.*** *(a) A series of AlN 0002 RC measurements taken at different positions with 1-mm steps in the range of 4 mm along the X direction. (b) Projection of the maximum peak in each RC profile on the (ω, X) plane: Δω is the difference between incident angles of X-ray beam at the positions X = −2 and 2 mm*

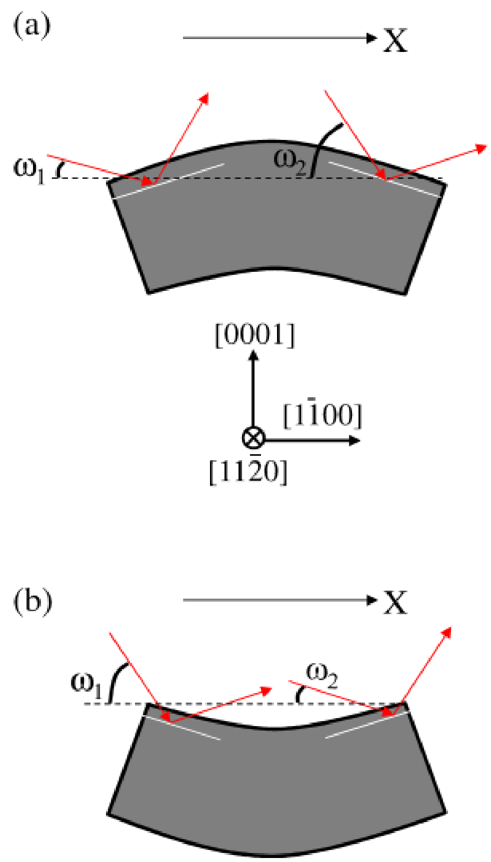


***Figure 4.*** *Schematic for determining the curvature of the thick AlN film in the X direction*

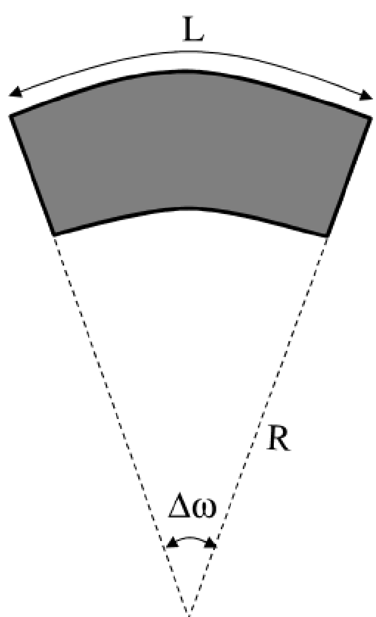


***Figure 5.*** *Schematic for determining the curvature radius of the thick AlN film in the X direction*

Here, *L* is the length probed by the X-ray beam on the film surface along the $[1\bar{1}00]$ direction, i.e., 4 mm. $\Delta\omega$ is the difference between incident angles of X-ray beam at the positions X = −2 and 2 mm, i.e., 0.07° as determined by the result shown in Figure 3(b). As a result, the curvature radius *R* is estimated to be 3.1 m.

## 4. Conclusion

The crystallographic curvature of the thick AlN film grown on the trench-patterned α-$Al_2O_3$ template was determined by performing a series of X-ray rocking curve measurements for AlN 0002 reflection at different positions across the AlN film surface. The results clarify that the AlN film is convexly bent along the $[1\bar{1}00]$ direction. The convex curvature of the AlN film is due to the presence of compressive strain in this direction.

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